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CARBON TREATMENT OF OTTO FUEL WASTEWATER — A LABORATORY STUDY

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24 MARCH 1977

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CARBON TREATMENT OF OTTO FUEL WASTEWATER - A LABORATORY STUDY: FINAL REPORT FOR 1 JULY 1972 TO 30 JUNE 1973

By

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W. F. Newton

ABSTRACT: Activated carbon has been found to be effective in removing Otto Fuel from water. Preliminary pilot runs using Filtrasorb 400 show a carbon cost of 1.8¢/gallon of wastewater treated as compared to 43¢/gallon for disposal by incineration. A carbon adsorption stage will be utilized in the design of an Otto Fuel Wastewater Treatment Plant. A prototype wastewater system will be the topic of a future report.

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INTRODUCTION

Many of the industrial operations at shore installations generate significant quantities of wastewater. One of these operations involves torpedo turnabout which creates an ordnance problem unique to the Navy. After a torpedo has been subjected to an exercise run, it is retrieved and returned to a Naval torpedo facility where it is overhauled, refueled, and readied for future service. Large volumes of wastewater are generated contaminated with oil, solvent, cyanide, particulates, and Otto Fuel (OF).

Executive Order 11752 requires that the quality of a wastewater being discharged conform to Federal and local regulations. Hence, environmental agencies have promulgated regulations that prohibit discharging the wastewater to the environment without first providing proper treatment to remove the contaminants to a permissible level.

In the absence of suitable treatment facilities, the wastewater from the operation has been disposed of either by incineration or in a landfill. Both of these methods are costly. It costs about six cents per gallon to transport wastewater which may generally contain no more than 0.5% of contaminants; and an estimated 500 gallons of #2 fuel oil is required to burn 1000 gallons of wastewater. A logical and also economical solution to this disposal problem is to concentrate the impurities first, then dispose of the resulting waste residues by incineration or by some other appropriate means.

According to a survey by Rosenblatt, Lauterback and Davis(1), several methods for treating nitrobody waste are possible, one of which is adsorption on activated carbon. Historically the use of activated carbon is known for removing taste and odor producing substances from water. However, the utility of activated carbon has been expanded to include the removal of explosive materials such as TNT, RDX, nitromethane, and nitroethane from industrial wastewater. Investigators have reported the use of activated carbon for the removal of TNT from waste streams in demilling operations, the manufacture of TNT, and loading and packing operations. Granulated activated carbon has been used to treat TNT contaminated water at Joliet(2) and Iowa Army Ammunition Plants. Thus there seemed to be a reasonable assurance that activated carbon might also succeed in the treatment of wastewater from the manufacture of nitrate esters, or of Otto Fuel contaminated water from the torpedo turnabout operation.

DISCUSSION

A. General

The objective of this study was to demonstrate the feasibility of using activated carbon for removing contaminants, especially Otto Fuel from wastewater generated in overhauling and refueling the MK46 and MK48 torpedoes. The fabrication, testing and evaluation of a prototype wastewater system will be discussed in a future report.

Generally the wastewater from the work areas in the shops is collected through floor drains and discharged directly to a sump. The contaminants consist mainly of flushing and lubricating oils, cleaning solvents, cyanides, particulates such as metal chips, wood fragments, and insects, and of course Otto Fuel. Otto Fuel is a liquid monopropellant used for the propulsion of Torpedoes MK46 Model 1 and MK48 Model 1. It is composed of a nitrate ester in solution with a desensitizing agent and a stabilizer. The nitrate ester, propylene glycol dinitrate (PGDN), is the energetic ingredient and is considered explosive by nature. Therefore, great care and sufficient forethought must be exercised when handling waste materials contaminated by Otto Fuel. Compositional changes through handling or unpredicted behavior caused by contaminants can introduce risks having undesirable consequences.

B. Preliminary Evaluation of Activated Carbon

Before determining the batch isotherms of several prospective activated carbons, two simple experiments were performed. The purpose was to determine the ability of activated carbon to remove Otto Fuel dissolved in water to an acceptable low level, and its adsorptive capacity. In the absence of a maximum concentration limit for Otto Fuel in wastewater, an arbitrarily chosen value of 1 ppm was used.

In the first experiment Filtrasorb 400, a granular bituminous carbon marketed by Calgon was mixed with distilled water previously saturated with Otto Fuel at room temperature. After a 1 hour contact period, the carbon was removed and the filtrate tested for nitrate using the diphenylamine(DPA) spot test. A negative test response was obtained. This test has a limit of detection of 0.5 ppm in the absence of interferences. In the second experiment, distilled water saturated with Otto Fuel was passed through a 1/2" diameter column containing 10 grams of granular Filtrasorb 400. The DPA test was used to monitor the effluent for the presence of Otto Fuel (actually PGDN). The addition of the "wastewater" was continued until a positive DPA test was obtained on the effluent. Elemental analysis showed that 1.18 grams of Otto Fuel had been adsorbed by the carbon. An operational loading capacity for FS400 has been calculated to be 11.8%.

The information gained from these experiments was sufficient to conclude that activated carbon can be used for treating Otto Fuel contaminated wastewater.

C. Adsorption Isotherms

The next step was to select a suitable carbon from among the many available commercially. Not all granular activated carbons are suitable for treating a particular type of wastewater. However, Freundlich isotherms may be used to get a general idea of how effectively a carbon will adsorb impurities present in the wastewater. Hence, batch adsorption isotherms were determined for several activated carbons. The adsorbents are listed in Table I and the test procedures used are given in Appendix A.

Table I

Adsorbents Evaluated

Trade Name	Manufacturer
Columbia	Union Carbide
Cullar D	Culligan, Inc.
Cullar F	Culligan, Inc.
Darco	ICI America, Inc.
Filtrasorb 400	Calgon Corp.
Norit A	R. W. Greeff
Nuchar C-190-N	Westvaco Corp.
Witco 256	Witco Chemical Corp.

Adsorption isotherm data for each of the carbons are given in Appendix B. These data are plotted on logarithmic paper giving the familiar Freundlich adsorption isotherms shown in Figure 1. The equilibrium concentration (C_e) of Otto Fuel is shown along the abscissa and the quantity of Otto Fuel removed per unit weight of adsorbent ($\frac{x}{m}$) is shown along the ordinate.

The empirical Freundlich equation may be expressed as follows:

$$x/m = K\, C_e^{\frac{1}{n}}$$

where m = weight of carbon
x = amount of Otto Fuel adsorbed
$\frac{x}{m}$ = weight of Otto Fuel adsorbed per unit weight of adsorbent.
C_e = Equilibrium concentration of Otto Fuel in solution after adsorption.
C_o = Concentration of Otto Fuel in influent.
K, n = Empirical constants.

The $\frac{x}{m}$ value represents the relative efficiency or adsorptive capacity, which is used in estimating how much carbon will be required for a particular application. Usually the material with the isotherm exhibiting the greatest $\frac{x}{m}$ value at C_o is preferred. Except for Norit A, the shallow slope of the isotherm for each carbon shows that dilute concentration of Otto Fuel is adsorbed at a relatively high weight capacity. In other words there is little change in the $\frac{x}{m}$ value as the concentration of Otto Fuel varies. On this basis, the data in Table II clearly show FS400 and Witco 256 are superior to the other condidates. The FS400 was selected to be used in the Otto Fuel wastewater studies.

Table II

Relative Efficiency of Adsorbents

Trade Name	$\frac{x}{m}$ at C_o (C_o=2000)
Columbia	.690
Cullar D	.478
Cullar F	.559
Darco	.430
Filtrasorb 400	.935
Norit A	.770
Nuchar C-190-N	.768
Witco 256	.917

It should be noted that these isotherms had been prepared from aqueous solutions of Otto Fuel that were free of impurities. In reality, the wastewater in a sump invariably contains numerous impurities which could affect the adsorptive properties of FS400. Hence, before proceeding to the column studies, Freundlich isotherms were determined using the actual wastewater from the sumps of the MK46 and MK48 torpedo turnabout operation. The isotherm data is tabulated in Appendix C, and the corresponding isotherms are plotted in Figure 2. These isotherms indicate that impurities in the wastewater are somewhat detrimental to the performance of the carbon, but not enough so as to abandon the use of FS400. The maximum loading capacity was determined for granular FS400 adsorption of Otto Fuel from distilled water, the MK46 wastewater, and the MK48 wastewater. The results are shown in Table III

Table III

Maximum Capacity of Granular FS 400
With Various Otto Fuel Wastewater

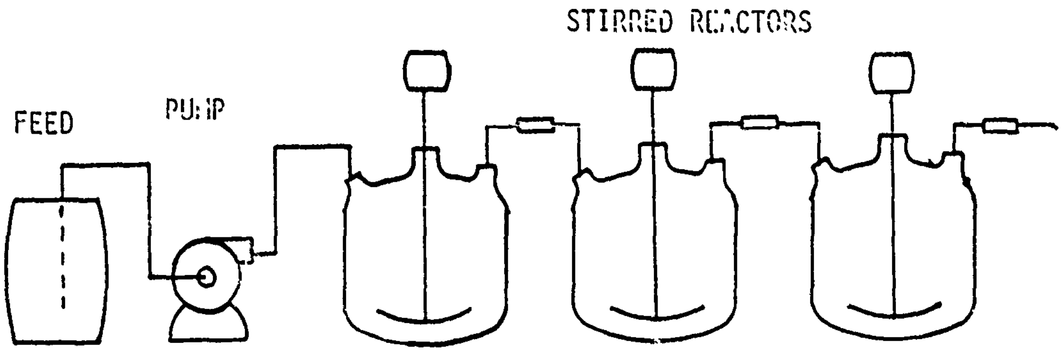
Wastewater	% Loading
MK46	51
MK48	55
Distilled water	66

The performance of activated carbon may be affected also by such factors as temperature, pH, and salinity. Adsorption isotherms using MK48 wastewater were prepared in which these factors were varied. The isotherm data is presented in Appendix D and the corresponding isotherms are plotted in Figures 3 through 5. The results in general show no substantial changes in loading capacity under changing conditions. Although the performance of the carbon appears slightly better at the lower temperature, there is no appreciable difference in capacity between 0°C and 43°C. The pH of the wastewater was adjusted to 3.3 with hydrochloric acid and to 9.5 with sodium hydroxide. The isotherms show that the loading capacity is not materially affected within the pH range selected. The salinity of the wastewater was adjusted to 1.27% and to 3.77% using artificial sea salts. As can be seen in Figure 5 the effect of salinity on adsorption efficiency is slight.

D. Methods of Adsorption

Carbon treatment may be applied as a batch contact process, or as a packed column. In the former case, enough carbon is added to a system to give the desired improvement and after a given contact time it is removed by settling and filtration. Theoretically most of the sorptive capacity of the carbon is subsequently used in the batch-contact process. In the packed column, a continuing stream of wastewater is passed through a granular carbon bed.

The batch contact process was investigated in the laboratory using the setup shown below:



The system was operated for an eight hour period. The wastewater processed in this experiment was prepared by dissolving Otto Fuel in tap water to the extent of 337 ppm. Upon terminating the test after eight hours of operation, the effluent from the first vessel contained 185 ppm Otto Fuel; none was found in the final effluent from the system. The loading of Otto Fuel on FS 400 was calculated to be 11.2%. This calculation was based on the adsorption of 16.4 grams of Otto Fuel by 130 grams of carbon which is the weight of FS 400 contained in the first two vessels. The amount of Otto Fuel adsorbed in the third vessel was insignificant. The scale factors (approx. 220:1) used are presented in Table IV.

Table IV

Batch Contact Process Data for Clean-Up of OF Wastewater

	Experimental Set-Up in Laboratory	Scaled Set-Up (calculated)
Carbon Charge per Vessel	65g	30 lbs.
Vessel Volume	1.0 L	55 gal.
Feed Flow Rate	15 ml/min	50 GPH
Total Volume Processed	48.6 L	2670 gal
OF Concentration in Feed	337 ppm	337 ppm
OF Concentration in Effluent from First Vessel	185 ppm	-
OF Concentration in Effluent from Second Vessel	< 10 ppm	-
OF Concentration in Effluent from Third Vessel	0 ppm	-

This mode of treatment has the advantage of simplicity of equipment required, the ease with which the carbon can be loaded in each stage as a slurry and the high efficiency because of maximum utilization of sorptive capacity. These advantages are countered by the high power consumption required to maintain the carbon granules in suspension, loss of carbon through size attrition and the additional equipment to filter contaminated fines from the final effluent.

Columnar adsorption was investigated using a glass column 5/8" diameter loaded with 103 grams FS 400 to a height of 36". The object was to get an idea of the loading profile of Otto Fuel on a Filtrasorb 400 column. Forty liters of tap water containing 1015 ppm of Otto Fuel was passed through the column using a peristaltic pump. At the termination of the test, the effluent contained 62 ppm Otto Fuel. The carbon was extruded from the column and sectioned, each section being analyzed for Otto Fuel by infrared spectroscopy. A total of 40.6 grams of Otto Fuel was found to be adsorbed on the carbon column. The loading profile data is tabulated in Table V which is in accord with the batch test data of Table III.

Table V

Otto Fuel Loading Profile on a Filtrasorb 400 Carbon Column

Column Section	Section Length inches	Otto Fuel %
1 (Top)	9	55
2	5	48
3	8	46
4	7	45
5 (Bottom)	7	13

The performance of the column was encouraging enough to investigate wastewater treatment on a larger scale.

E. Safety Study

Before beginning the scaled-up investigation, an assessment of the explosion or fire hazards had to be made because as pointed out earlier, propylene glycol dinitrate, the energetic component of Otto Fuel has an explosive character. The results of the safety tests performed are presented in Table VI. No exceptional hazards are seen provided the process conditions are kept within the limits established for neat OF, the temperatures are kept under 75°C, and the contaminated carbon is kept wet.

Table VI

Safety Data

Material	Start of Exotherm Reaction	Impact Sensitivity (5Kg Wt)	Vacuum Stability ml of gas evolved Ambient 24 hrs.	90°C 4 hrs	90°C 21 hrs
Neat OF	180°C	> 600 mm	.17	.09	.06
OF + FS400 1:1 (paste)	155°C	> 600 mm	.59	4.65	10.58
OF + FS400 wet w/H_2O	160°C	> 600 mm	-	-	-

F. Large Scale Packed Column Investigation

A large scale column test was run using the Calgon Pilot Filtrasorb System. The system consists of four five-inch diameter columns in series. Each was packed with about 20 pounds of FS400 giving an average bed depth of 3.5 feet per column or a measured total of 13.5 feet. The unit was operated in the down flow mode under conditions considered likely to be encountered in the field.

For this test, 250 gallons of wastewater (in 55 gallon steel drums) were obtained from a naval ordnance weapons test facility (Complex 30) at Cape Kennedy, Florida. The undissolved Otto Fuel, solvent and other impurities were removed from the bottom of each drum and used to prepare an additional 2000 gallons of wastewater. Visual examination of the effluent showed no evidence of oil being present. A test for cyanide using the specific ion electrode proved negative. The operating data and the test results are given in Table VII.

Table VII

Operating Data and Test Results for Otto Fuel Wastewater Treatment in a Column

No. of Columns	4
Total Weight	80 lbs.
Bed Diameter	5 in.
Bed Depth	13.5 ft
Bed Area	.137 ft^2
Bed Volume	1.84 ft^3
System Pressure (avg.)	16 psig
Operating Time	75 hrs
Flow Rate	30 GPH or 3.7 GPM/ft^2 bed area
Wastewater Processed	2250 gal.
Otto Fuel concentration in feed	260~400 ppm

Test Results

Otto Fuel adsorbed by FS400	6.6 lbs.
Loading Ratio (avg. for first three columns)	8.25%
Lbs. of carbon/gal of w/w processed	.036 lbs
Carbon cost/gal (carbon cost 50¢/lb)	1.8¢
Cost of Carbon/lb of Otto Fuel removed	$6.06
Otto Fuel concentration in final effluent	<1.0 ppm[a]
pH	6.5~7.5

a (DPA spot test)

Although the carbon costs 1.8 cents per gallon of wastewater treated this is still encouraging when compared to the 43 cents per gallon for disposal by incineration (See Appendix E).

The contaminated carbon still wet with water was left standing for a few days after the test was terminated. Due to the biological action under the anerobic conditions present, the indisputable odor of hydrogen sulfide was detected emanating from the columns. The greatest biological activity was observed in the final column. Gas evolution was measured as averaging 2 liter/day. It is interesting to note that the greatest biological activity was also observed in effluent samples that contained oil only; while little or no activity was detected in water samples that contained Otto Fuel. Perhaps Otto Fuel has an inhibitory effect on biological activity which might be the reason why the last column, being relatively free of Otto Fuel, had the greatest biological activity. If intermittent use of a carbon adsorption unit is planned in the field, operation problems are almost certain to develop because of this biological action. Of course, the columns could be sized for a short service time, or the wastewater could be pretreated to remove undissolved oil droplets.

Conclusion

1. Filtrasorb 400 has been found to be effective in removing Ottl Fuel from wastewater.

2. A prelimin-ry large-scale run on the Calgon Filtrasorb Pilot Plant shows a carbon cost of 1.8¢/gallon of wastewater treated as compared to 43¢/gallon for disposal by incineration.

3. A carbon adsorption stage will be utilized in the design of an Otto Fuel wastewater treatment plant.

References

(1) Rosenblatt, D., G. Lauterbach, and G.T. Davis, "Water Pollution Problems Arising from TNT Manufacturer - A Survey", EASP 100-94, Research Laboratories, Edgewood Arsenal, Maryland, March 1971.

(2) Hebert, Paul V., "Water Quality Engineering Special Study No. 24-024-72/73 Wastewater Study, Joliet Army Ammunition Plant, Joliet Illinois, 5019 June 1972", U.S. Army Environmental Hygiene Agency, Edgewood Arsenal, Maryland.

Appendix A

Procedures for Freundich Isotherm

Each of the carbons to be treated were ground to pass through a 325 - mesh sieve, washed with distilled water, then dried in an oven at 105°C for three hours. Weighed quantities of each of the pulverized carbons were added to flasks containing 1600~1800 ppm Otto Fuel dissolved in distilled water. After one hour of agitation at ambient temperature, the contents of each flask was vacuum filtered through a 0.45 micron glass filter. The residual Otto Fuel was extracted from 100 ml of each filtrate with three 10 ml aliquots of methylene chloride. The methylene chloride extract was passed through phase paper to remove the water and the Otto Fuel content measured by infrared spectroscopy at 1645 cm^{-1} on a Perkins Elmer Model IR 180.

Appendix B

Isotherm Data for Activated Carbons and Otto Fuel - Water Mixtures

Carbon	Carbon, ml/l (m)	Residual OF, ppm (C_e)	OF adsorbed, ppm (x)	Adsorptivity ($\frac{x}{m}$)
COLUMBIA	0	1680	0	-
	808.8	1146	534	.660
	1210.8	987	693	.572
	1608.0	671	1009	.627
	2005.2	524	1156	.577
CULLAR D	0	1697	0	-
	807.2	1314	383	.474
	1200.8	1145	552	.460
	1610.8	933	764	.474
	2010.8	772	925	.460
CULLAR F	0	1750	0	-
	804.8	1308	442	.549
	1211.6	1088	662	.546
	1601.2	850	900	.562
	2028.0	672	1078	.532
DARCO	0	1716	0	-
	808.0	1411	305	.377
	1226.8	1261	455	.371
	1624.0	1047	669	.412
	2020.0	988	728	.360

Appendix B (cont)

Isotherm Data for Activated Carbons and Otto Fuel - Water Mixtures

Carbon	Carbon, mg/l (m)	Residual OF, ppm (C_e)	OF adsorbed, ppm (x)	Adsorptivity ($\frac{x}{m}$)
FS 400	0	1725	0	-
	822.0	1062	663	.807
	1208.0	758	967	.801
	1608.0	484	1241	.772
	2026.0	292	1433	.707
NORIT A	0	1606	0	-
	801.2	1182	424	.530
	1204.0	980	626	.520
	1612.4	924	682	.423
	2016.8	760	846	.419
NUCHAR C-190 N	0	1716	0	-
	811.2	1190	526	.648
	1202.4	938	778	.647
	1606.4	746	970	.606
	2020.0	570	1146	.567
WITCO 256	0	2330	0	-
	804.0	1606	724	.900
	1207.0	1245	1085	.899
	1602.0	890	1440	.899
	2002.0	600	1730	.864

Appendix C

Adsorption Isotherm Data for FS 400 and MK46 and MK48 Otto Fuel Wastewater

Wastewater	Carbon, mg/l (m)	Residual OF, ppm (C_e)	OF Adsorbed ppm (x)	Adsorptivity ($\frac{x}{m}$)
MK 46 Torpedo Operations	0	1960	0	-
	800.4	1453	507	.633
	1208.0	1245	715	.592
	2409.6	425	1435	.596
	3204.8	134	1826	.590
	4000.0	97	1863	.459
MK48 Torpedo Operations	0	2823	0	-
	814.4	2183	640	.786
	1200.4	1815	1008	.840
	2408.0	947	1876	.779
	3210.0	466	2357	.734
	4012	275	2548	.635
	5260	125	2698	.513

Appendix D

Adsorption Isotherm Data for FS 400 - MK48 Otto Fuel Wastewater at Different Temperatures, pH's and Salinities

Variable	Carbon, mg/l (m)	Residual OF, ppm (C_e)	OF Adsorbed ppm (x)	Adsorptivity ($\frac{x}{m}$)
Temperature				
0°C	0	2758	0	-
	800.0	1940	818	1.02
	1602.0	1221	1537	.960
	2404.0	680	2078	.864
	4000.0	130	2628	.657
43°C	0	2680	0	-
	800.0	2000	680	.850
	1602.0	1303	1377	.860
	2406.0	730	1950	.810
	4008.0	188	2492	.623
pH				
3.3	0	2712	0	-
	807.0	2164	548	.679
	1606.0	1581	1131	.704
	2406.0	1091	1621	.674
	4012.0	340	2372	.591
9.5	0	2619	0	-
	804.0	2069	550	.684
	1606.0	1492	1127	.702
	2401.0	944	1675	.697
	4010.0	244	2375	.592
Salinity				
1.27%	0	2823	0	-
	814.4	2183	640	.786
	1200.4	1815	1008	.840
	1598.0	1534	1289	.807
	2408.0	947	1876	.779
	4012.0	275	2548	.635
3177%	0	2686	0	-
	806.4	2028	658	.816
	1606.0	1616	1070	.666
	2406.0	855	1831	.761
	4008.0	178	2508	.626

Appendix E

NAVTORPSTA, Keyport
Incineration Cost/Gallon

13 Oct 73 thru 19 Nov 73
Based on 24 Hours Operation

Waste

Waste burned = 43,309 gal

Incineration

Labor 1099 hrs x $9.38/hr = $10,306.34

a. #2 Fuel Oil
26,834 gal x $.21/gal = 5,635.14

b. Propane
60 gal x $.25/gal = $15.00

c. Electricity Negligible
$15,956.48

Transportation

Labor 261 hrs. x $9.38/hr = $2,445.90

Costs

Incineration $15,956.48/43,309 gal = $.37/gal
Transportation $2,445.90/43,309 gal = $.06/gal

Total = $.43/gal

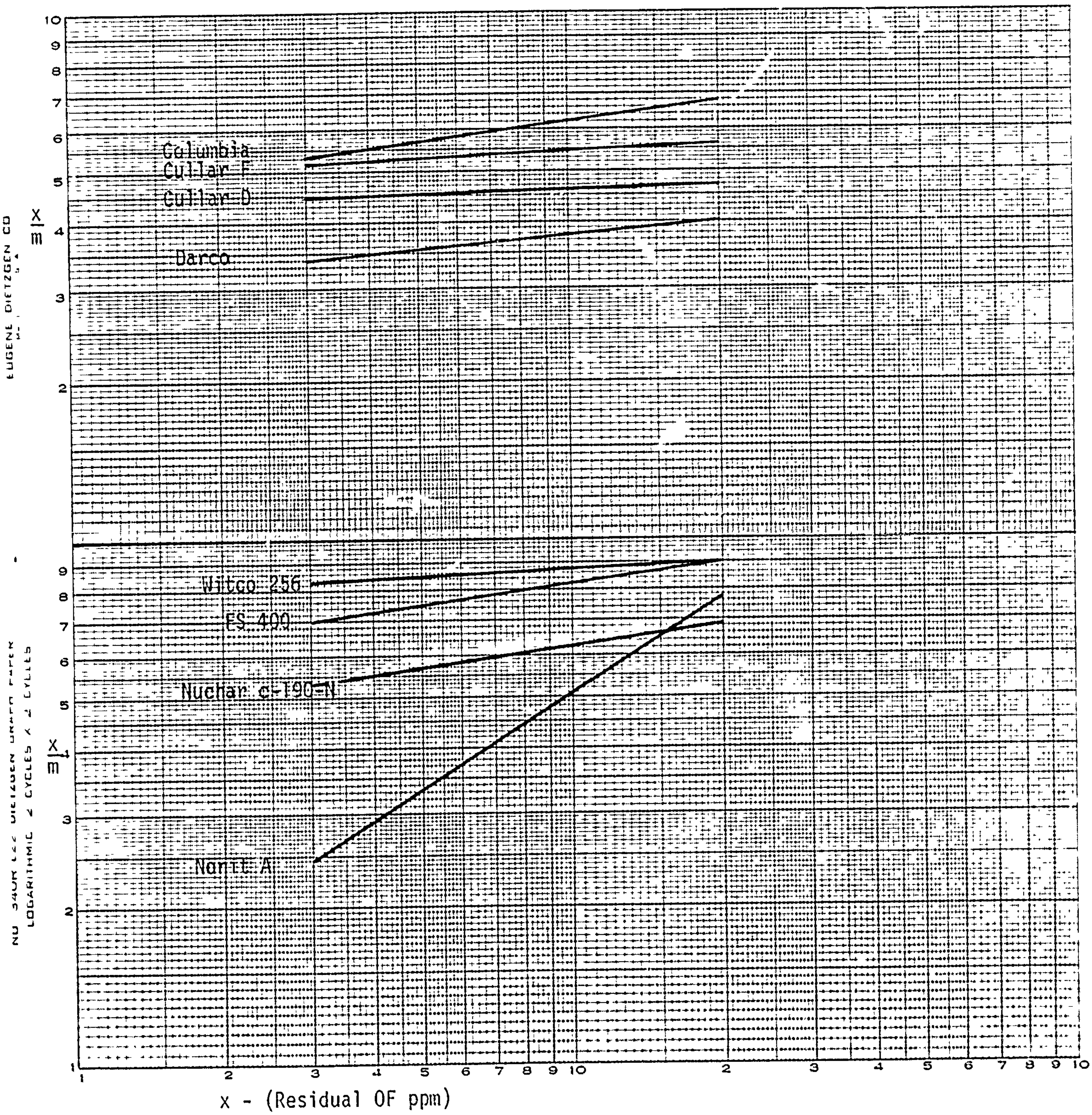
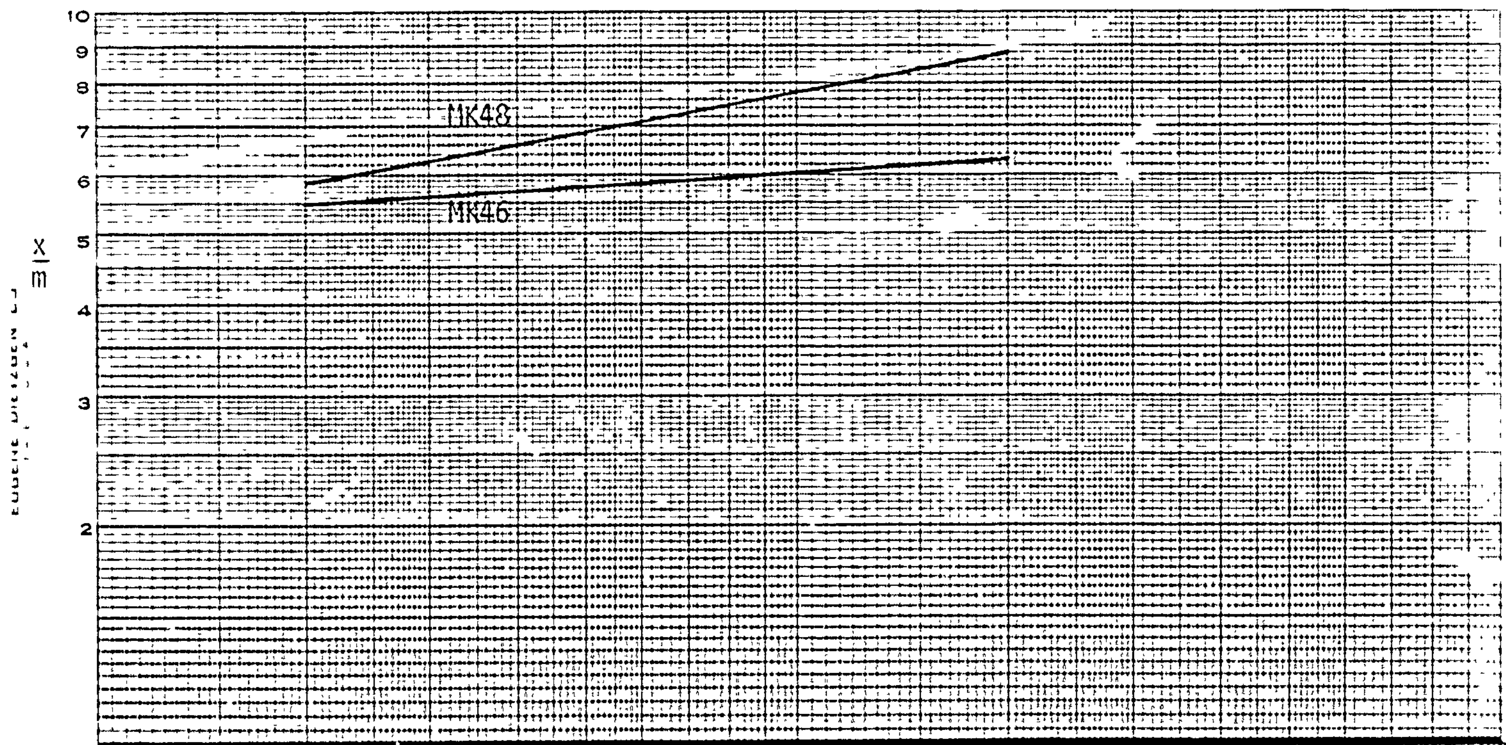


Figure 1: Adsorption Isotherms for Activated Carbon and Otto Fuel - Water Mixtures.

Figure 2: FS 400 Isotherms for MK46 and MK48 Otto Fuel Wastewater



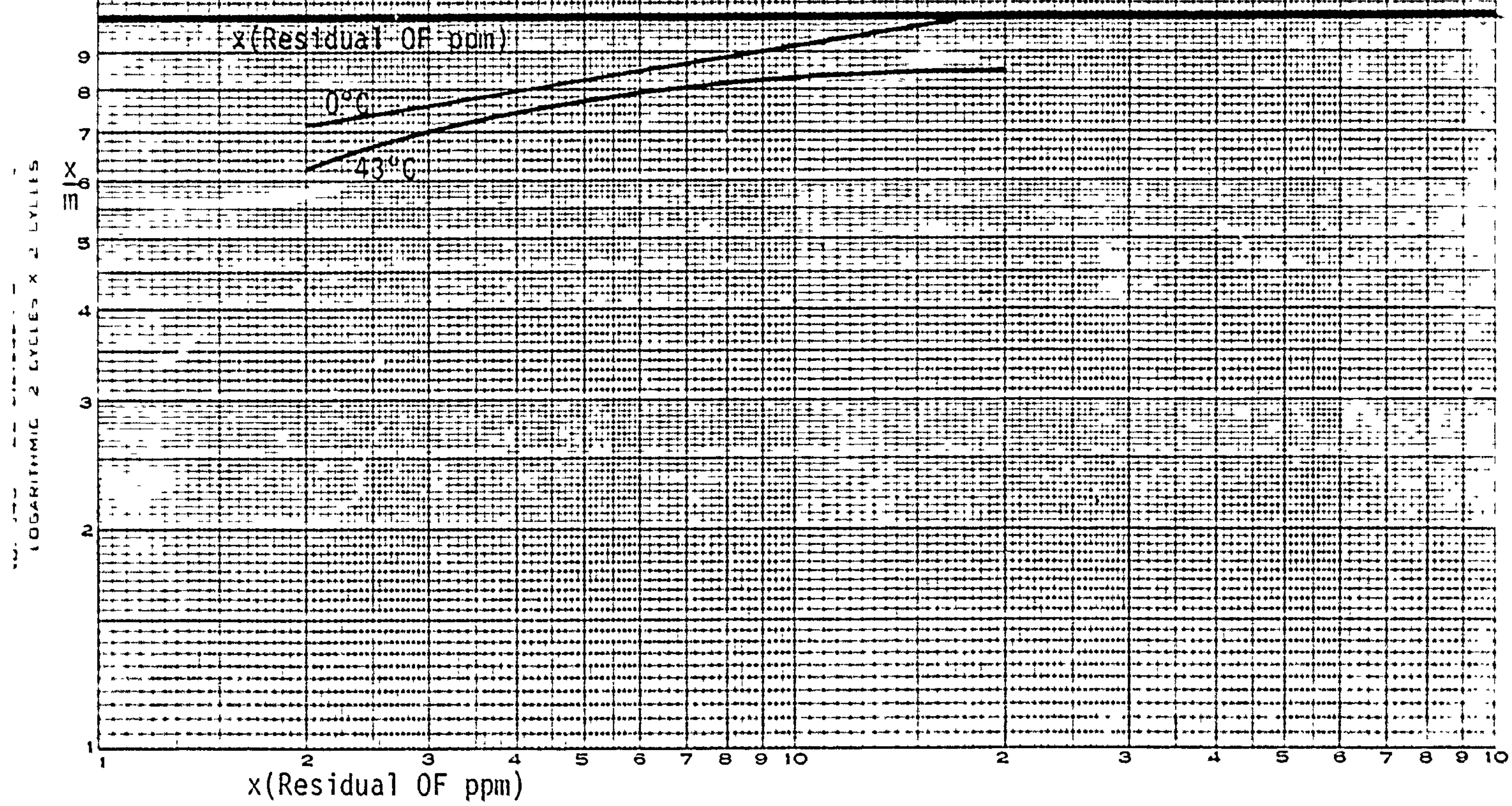
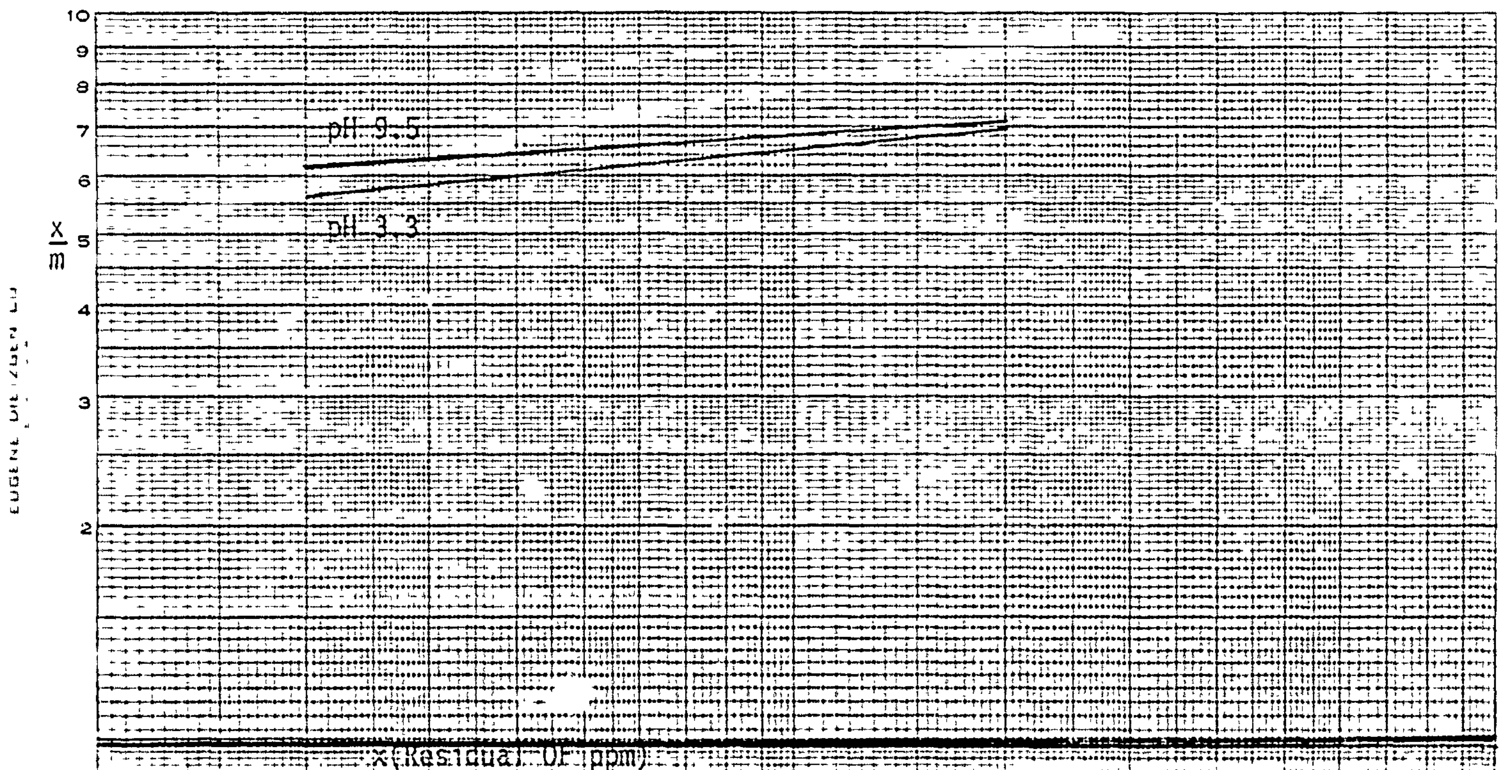


Figure 3: Effect of Temperature on FS 400 - MK48 Wastewater (Isotherm)

Figure 4: Effect of pH on FS400 - MK48 Wastewater (Isotherm.)



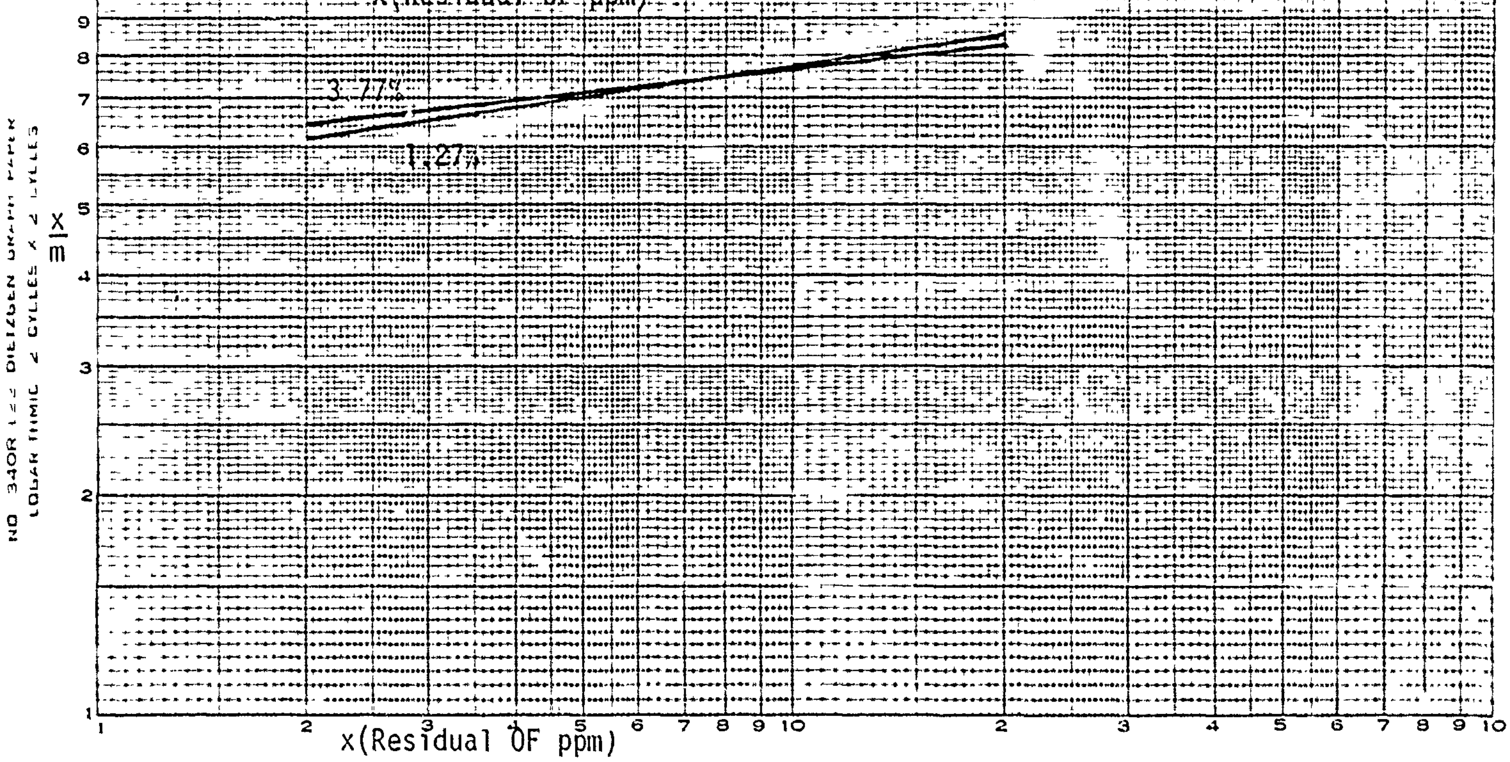


Figure 5: Effect of Salinity on FS 400 - MK48 Wastewater (Isotherm.)

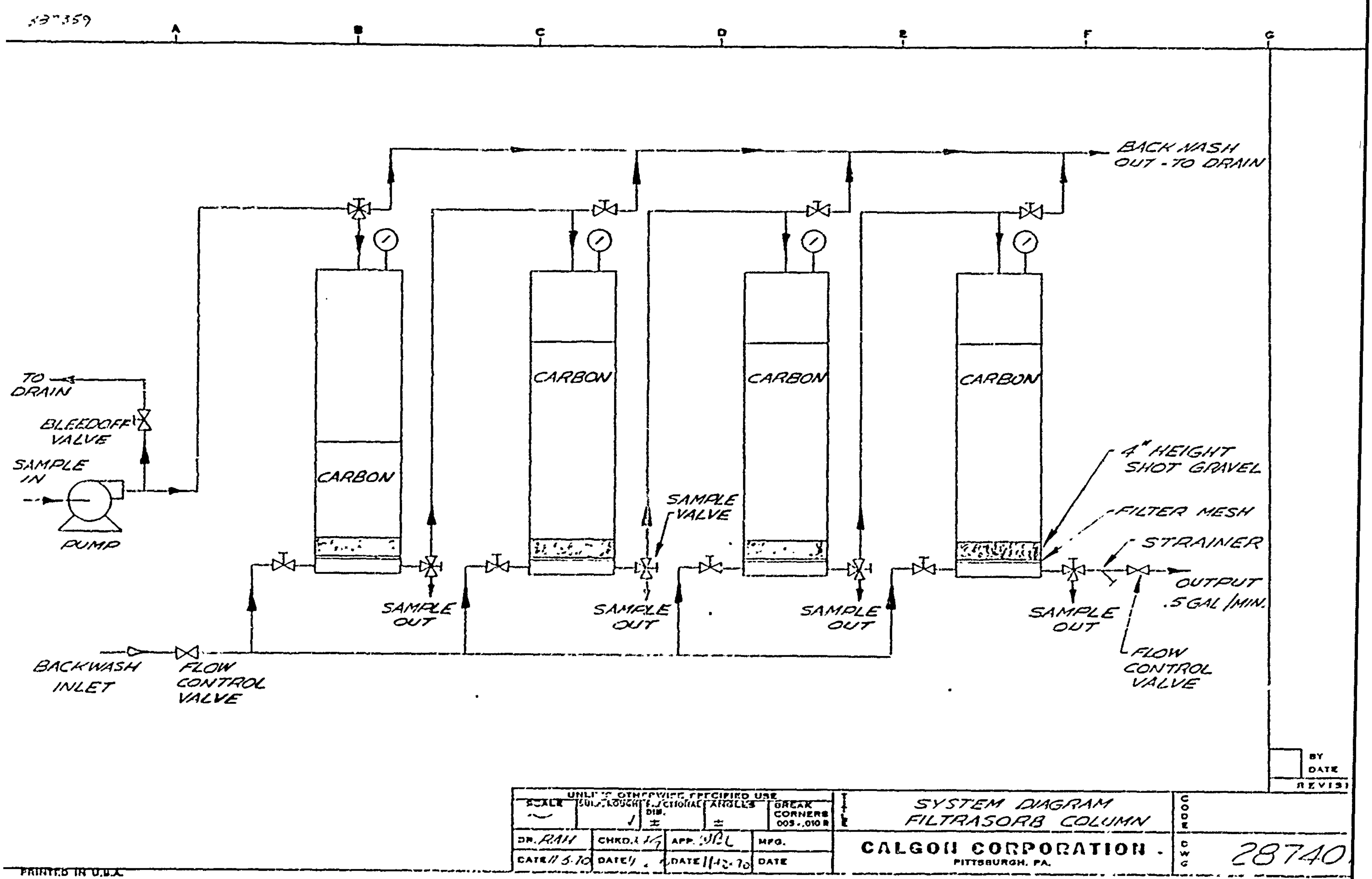


FIGURE 6: Calgon Pilot Filtrasorb System

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